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
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 Dust free particulate enzyme formulation.

 Disclosed is a method for the production of dust free, enzyme containing particles. The method involves coating a hydratable core particle with the enzyme in a fluidized bed reactor and then applying an overcoating of a film-forming macromolecular material to the enzyme coated core.

EP 0 193 829 A2

1     DUST FREE PARTICULATE ENZYME FORMULATION

BACKGROUND OF THE INVENTION

          This invention relates to a procedure for making dry and dust free enzyme granules particularly useful  
5 for use with laundry detergents. The manufacture of enzymatic washing and cleaning agents by incorporating powdered, highly active enzyme concentrates by mixing them with common cleaning agents is well known. The washing agents manufactured in this  
10 manner tend to form enzyme dusts which can cause dermatologic damage both to the manufacturer and the consumer of the enzyme powder containing washing composition.

          Various enzyme formulations and processes for  
15 these preparations have been developed in an effort to alleviate the dusting problem. For example, German AS 2' 37 042 discloses a process in which an extrudable enzyme containing formulation is extruded through a die onto the revolving plate of a spheron-  
20 izing device to form spherical particles of the enzyme containing formulations which are optionally coated with a material designed to prevent dusting.

1       In U.S. Patent No. 4,087,368, there is disclosed  
an enzyme granule formulation in which rods or  
spheres of an enzyme in admixture with magnesium  
alkyl sulfate and ethylene oxide are provided.

5       U.S. Patent No. 4,016,040 discloses a method for  
the preparation of free-flowing substantially dust  
free, spherical enzyme containing beads prepared by  
blending a powdered concentrate of the enzyme with a  
binder in molten form and spraying droplets of the  
10 blend through a spray nozzle into cool air to solid-  
ify the droplets and form the beads.

      In U.S. Patent No. 4,242,219, there is claimed a  
process for the preparation of enzyme containing  
particles prepared by mixing the dry enzyme with a  
15 hydrophilic organic cohesive material, a building  
agent and a moisture regulating agent and mechan-  
ically dividing it into particles of the desired size  
and shape which are then coated with a water repel-  
lent material.

20       Another type of granular enzyme formulation is  
described in U.S. Patent No. 4,009,076. This formu-  
lation is prepared by mixing the dry enzyme with a  
solid non-viable substance and optionally a cohesive  
organic material as binder to form an enzymatically  
25 active core. An enzyme slurry containing the cohe-  
sive organic material can be sprayed onto, for  
example, sodium tripolyphosphate in a mixer or an  
enzyme powder can be mixed with the sodium tripoly-  
phosphate and the cohesive organic material sprayed  
30 onto it with subsequent extrusion through a die. The  
enzyme containing granule is sprayed with an aqueous

- 1 solution containing a plasticized organic resin and  
then dried.

A process is described in DDR Patent No. 0 151  
598 in which sodium tripolyphosphate is sprayed with  
5 an aqueous enzyme solution and agglomerated in a  
cyclone apparatus. The agglomerates are removed from  
the cyclone apparatus while still wet and placed in a  
mechanical blender with a drying detergent formula-  
tion and intensively mixed.

- 10 In British Patent No. 1,483,591, there is  
described a process for coating water soluble or  
water dispersible particles, including enzyme parti-  
cles, using a fluid bed reactor. This reference  
involves a dust free coating technique for enzyme  
15 particles which have been granulated by other pro-  
cesses such as prilling or spheronizing whereas the  
process of this disclosure applies an active layer of  
enzyme onto an inert core.

SUMMARY OF THE INVENTION

- 20 The present invention is a method for the  
production of dust free enzyme containing particles.  
The method comprises the steps of:

- a) introducing a particulate, hydratable core  
material into a fluid bed dryer and maintaining  
25 the core particles suspended in the dryer's  
reaction chamber;

- 1 b) providing an aqueous slurry of a water soluble  
or dispersible enzyme and applying the enzyme to  
the surface of the core particles by spraying  
the slurry onto them while they are suspended in  
5 the reaction chamber to leave residual, dried  
enzyme coated on the core particles in an amount  
sufficient to provide the desired enzyme  
activity;
- 10 c) spraying a solution or dispersion of a macro-  
molecular, film-forming, water soluble or water  
dispersible coating agent onto the enzyme coated  
core material while it is still suspended in the  
reaction chamber and drying the solvent to leave  
15 a continuous layer of the film-forming material  
on the enzyme coated core particle to provide  
the desired dust free enzyme containing parti-  
cle.

Also included within the scope of this invention  
are the enzyme containing particles prepared by this  
20 process.

#### DESCRIPTION OF THE INVENTION

The method of the present invention is carried  
out in a fluid bed dryer. Typically, such devices  
comprise a dryer consisting of a circular product  
25 chamber that has a porous grid on the bottom and is  
open on the top to be put up against a conical shaped  
expansion chamber of a larger diameter than the

1 circular product chamber. In operation, as the  
velocity of air passing up through the chamber is  
increased, a point is reached where particles resting  
on the porous grid are suspended in the air flow as a  
5 fluid, hence the terms "fluidization" and "fluid bed  
dryer". The particles are lifted by the upward force  
of the air out of the product chamber into the  
expansion chamber where the air expands and the  
upward force per unit of area is reduced. This  
10 allows the particles to fall back into the product  
chamber and start the cycle over.

The initial step in the method involves intro-  
ducing a particulate, hydratable core material into  
the reaction chamber of the fluidized bed reactor and  
15 suspending the particles therein on a stream of air.  
The core particles are preferably of a highly hydrat-  
able material, i.e. a material which is readily  
dispersible or soluble in water. The core material  
should either disperse (fall apart by failure to  
20 maintain its integrity) or solubilize by going into a  
true solution. Clays (bentonite, kaolin),  
non-pareils and agglomerated potato starch are  
considered dispersible. Non-pareils are spherical  
particles consisting of a solid core that has been  
25 rounded into a spherical shape by binding layers of  
powder to the core in a rotating spherical container.  
The non-pareils used in the examples which follow  
have a sugar (typically sucrose) crystal core less than  
0.3 mm (-50 mesh on the U.S. Standard Sieve Series) that was  
30 rounded by binding layers of corn starch onto the  
core using sugar as a binder. The sugar used for

- 1 binding was dissolved in water (50% w/w) and sprayed  
onto a mixture of sugar and corn starch while they  
were being rotated in a 167,54 cm (66 inch) Groen Stainless Steel  
Rotating Pan which were then heated to drive off the  
5 water. When the crystals had been rounded into approximately  
0,85 mm to 0,25 mm (-20 mesh to 60 mesh) spheres, they were dried  
and sieved whereupon the >0,85 mm, <0,25 mm (-20 mesh +60 mesh)  
fractions were put back into the rotating pan and  
heated. They were then coated with a layer  
10 (approximately 10% w/w) of dextrin from an aqueous  
solution (50% w/w) that was sprayed onto the spheres  
while heating to drive off the water. The finished  
product was again sieved to 0,85 mm to 0,25 mm (-20 mesh +60 mesh).  
Salt particles (NaCl crystals, NaCl rock salt,  $\text{NaHCO}_3$ ) are  
15 considered soluble. More particularly, core  
particles can be non-pareils of a salt crystal,  
starch and a sugar solution or a sugar crystal,  
starch and a sugar solution with or without a final  
coat of dextrin or a confectionary glaze. Also  
20 suitable are agglomerated trisodium citrate, pan  
crystallized NaCl flakes, bentonite granules and  
prills, bentonite/kaolin/diatomaceous earth disk  
pelletized granules and sodium citrate crystals. The  
core particle is of a material which is not dissolved  
25 during the subsequent spraying process and is of a  
particle size of from 150 to 2,000 microns (100 mesh  
to 10 mesh on the U.S. Standard Sieve Series) in its  
longest dimension.

- Enzymes suitable for use in this method are  
30 those which are soluble or dispersible in an aqueous  
media and from which the water can be removed to

- 1 leave a residual layer of enzyme on the surface of  
the core material. Suitable enzymes include, for  
example, proteases (bacterial, fungal, acid, neutral  
or alkaline), amylases (alpha and beta) and lipases  
5 whose water solutions or dispersions are prepared by  
dispersing or dissolving a precipitated enzyme cake  
in water using vigorous agitation. Typically, the  
enzyme precipitate is dissolved or dispersed at a  
level of 15% to 30% solids (w/w) of which 100% down  
10 to about 30% is enzyme with the remaining solids  
comprising metallic salts, binders, plasticizers and  
fragrances. The dispersion, including any optional  
binders, metallic salts, stabilizers or fragrances  
must have a viscosity low enough (typically 10 to  
15 5,000 cps at room temperature) to be pumped and  
atomized for effective spray coating. The enzyme is  
applied to the surface of the core material by  
fluidizing the core particles in a flow of air  
whereupon a solution containing the enzyme and  
20 optionally other solids is then atomized and sprayed  
into the fluidized bed. The atomized droplets  
contact the surface of the core particles leaving a  
film of the solids adhering to the surface of the  
particles when the water is evaporated.
- 25 When sufficient enzyme is applied to the core  
particles to provide the desired enzyme activity, the  
enzyme coated particles, while still suspended in the  
reaction chamber of the fluidized bed reactor, are  
coated with a uniform layer of a water soluble or  
30 water dispersible, macro-molecular, film-forming  
coating agent. This is accomplished in a manner



1 similar to that used for application of the enzyme  
coating. Suitable film-forming agents include, for  
example, fatty acid esters, alkoxyated alcohols,  
polyvinyl alcohols, ethoxyated alkylphenols and more  
5 specifically, polyethylene glycols (MW 1,000 to  
8,000), linear alcohol alkoxyates (MW 1,450 to  
2,670), polyvinyl pyrrolidone (MW 26,000 to 33,000),  
polymeric nonylphenyl ethoxyates (MW 1,975 to 4,315)  
and dinonylphenyl ethoxyate (average MW 6,900). The  
10 net result of the process is to provide an enzyme  
coated core particle having a continuous layer of the  
film-forming material on its surface to provide the  
desired dust free enzyme containing particle.

The dust free enzyme particles of the present  
15 invention can be used wherever enzymes are needed in  
an aqueous system. Thus, they can be used as addi-  
tives to detergent formulations, for removing gelatin  
coatings on photographic films to aid in silver  
recovery, in the digestion of wastes from food  
20 processing plants for nitrogen recovery, in denture  
cleansers for removing protein bound stains and as a  
processing aid in waste water treatment.

The method of practicing the invention is  
further illustrated by the following examples where  
25 all mesh sizes are on the U.S. Standard Sieve Series,  
and the dryer is a Uni-Glatt laboratory model fluid  
bed dryer with variable air temperature and flow  
through the bed. The device has a 15.24 cm (6 inch) Wurster  
insert which consists of a container 13.97 cm<sup>3</sup> (5-1/2") diameter  
30 by 16.51 cm (6-1/2") height) for the core material that fits  
against the bottom of the device's expansion chamber.

- 1 The plate on the bottom of the Wurster has holes in it to distribute the air through the bed with the holes in the center being of a larger diameter than the rest of the holes in the plate. A cylindrical
- 5 hollow tube (7cm (2-3/4) inches diameter by 15,24 cm (6 inches) length) called a partition is suspended above these larger diameter holes creating a higher air flow up through the partition than up around the outside of the partition. The air flow is adjusted based on the
- 10 quantity and density of the core particles so that the particles flow up inside the partition into the expansion chamber then fall back down outside the partition into the area with less air flow while the bed is kept fluidizing and drying. This difference
- 15 in air flow creates a circular upward and downward movement of the particles. The spray nozzle is installed at the bottom of the partition pointed upwards. This arrangement keeps the atomized liquid co-current with the motion of the cores being coated
- 20 and results in a smooth coating. The speed of the circular flowing motion of the cores is adjustable by regulating the amount of air going through the partition and the amount of air going around the outside of the partition. The droplet size of the
- 25 atomized enzyme solution spray is adjusted by adjusting the liquid pumping rate and the air pressure for atomization. The process can be accelerated by using counter current downward spray without using the Wurster column.
- 30 The height of the Wurster insert partition is adjustable vertically and was adjusted from 0,64cm (1/4 inch)

1 to 1.9 cm (3/4 inch) up from the bottom plate. When denser  
core materials are used, up to 3/4 of the holes  
outside the partition were blocked off to provide a  
higher linear velocity for the air to lift the  
5 particles up through the inside of the partition and  
maintain a smooth circulation of material through the  
spraying area. The total air flow was adjusted to  
get a good flow of cores through the partition and  
keep the bed outside the partition fluidized. Inlet  
10 air temperature was adjusted up to a maximum of 75°C  
so that the outlet as well as particle temperatures  
were below 50°C. Typical outlet temperatures during  
the coating process were 25°C to 40°C. The solids  
level of enzyme slurry sprayed in was 15% to 30% of  
15 the solution (w/w). Feed rate varied from 5 ml/min.  
to 20 ml/min. When a more soluble core material was  
used, a lower initial feed rate was essential to coat  
a layer of enzymes on the core before the feed rate  
was increased. Atomization air pressure ranged from  
20 1.0 to 1.5 bar. A typical dry weight gain of the  
core material after enzyme coating is 10% to 35%  
depending on the final activity desired. The enzyme  
coated core was further coated with a macro-  
-molecular, film-forming, water soluble or water  
25 dispersible coating agent to seal the enzyme from  
contact with the atmosphere or persons handling the  
particle. After application of the enzyme and  
protective coating, the typical total dry weight gain  
based on the weight of the core material after the  
30 dust free coating is 25% to 55%.

1        In the following examples, the core materials  
are either salt or non-pareils. Salt is totally  
soluble and water clear when dissolved and is inex-  
pensive as a core material. Being a solid crystal  
5 and not a multicomponent structure, it is less subject  
to breaking up during the coating process and the  
enzyme slurry can be sprayed at a faster rate.  
However, the salt particles being cubes make them  
more difficult to coat because there is a greater  
10 tendency for poor binding between the film and the  
core. Furthermore, enzyme coated salt crystals are  
more subject to film loss due to attrition from the  
corners of cubes striking the flat surfaces of  
others. This problem can be partially alleviated by  
15 adding binders or plasticizers to the enzyme slurry.  
Suitable materials include carboxymethyl cellulose,  
sodium alginate, collagen, polyethylene glycol and  
ethoxylated alkylphenols in an amount of from 1 to  
10% (w/w) of the total solids in the slurry. In  
20 addition, the flat surfaces provide larger areas of  
contact between particles which can cause agglomera-  
tion thereby inhibiting the flow characteristics of  
the coated salt particles. The non-pareils are  
spherical, can readily be coated with a continuous  
25 film and have less area of contact among particles  
thereby limiting agglomeration. The final spherical  
product has better flow characteristics than the  
cubic salt based enzyme product.

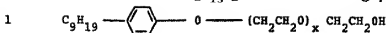
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EXAMPLE 1

Laboratory Fluid Bed Spray Coating  
of Alkaline Protease

Eight hundred and eighty-five grams of non-  
pareil particles (prepared by spraying a sugar solution onto  
sugar crystals which were coated with starch followed by a final  
coat of dextrin, less than 0,6 mm, but greater than 0,25 mm (-30 +60  
mesh) were charged to the Uni-Glatt device previously  
described and fluidized. An aqueous enzyme slurry  
with 16% dry solid at the detergent alkaline protease  
level of 650 DAPU/gm (DAPU = Detergent Alkaline  
Protease Unit) was fed into the dryer for coating at  
the rate of 8 ml/minute. A total of 716 g of enzyme  
slurry containing 115 g of enzyme solid was sprayed  
onto the particles.

The enzyme coated particles were further coated  
with a nonylphenol ethoxylate having an average  
molecular weight of 4315 marketed under the trademark  
Iconol NP-100 (BASF-Wyandott Corp.) by spraying 120g  
of its aqueous solution onto the particles in the  
Uni-Glatt device. The solution, which was 50% w/w,  
contained 60 g of the Iconol NP-100 and was sprayed  
at a rate of 8 ml/minute. Iconol NP is a nonionic  
chemical compound composed of a nonylphenol hydro-  
phobe and a polyoxyethylene group hydrophile all in  
one molecule. The material is represented by the  
structural formula:



with X being approximately 100 in the NP-100 material.

5      The coated particles were further cosmetically coated with 260 g of an aqueous solution containing 82 g (31.5% w/w) titanium dioxide and 27 g (10.4% w/w) Iconol NP-100 at the feed rate of 8 ml/minute.

A final total of 1116 g dust free particles was harvested with a final activity of 390 DAPU/g as determined by Detergent Alkaline Protease Units Procedure, Miles Laboratories, Inc. QA Procedure #ME400.23 available from Miles Laboratories, Inc., Enzyme Technical Service Department, P.O. Box 932, Elkhart, IN 46515. This test resulted in 100% mass balance yield and 96% of enzyme yield.

15      The Uni-Glatt operation conditions were as follows:

Air Regulation Flap Level	: Fully Open
Product Pressure Differential	: 0.5 Kilo-pascals
20    Outlet Air Pressure Differential:	200-250 mm Water
Atomization Air Pressure	: 1.5 Bar
Inlet Air Temperature Setting	: 60/64°C and 50-54°C
Outlet Air Temperature Range	: 30-40°C
25    6 inch Wurster Insert	
Clearance from Bottom Plate	: 0.64 cm (1/4 inch)
Angle setting	: 3 mm

1

EXAMPLE 2

Laboratory Fluid Bed Spray Coating of Both  
Alkaline Protease and Alpha-Amylase

In this run, the Uni-Glatt operating conditions  
5 were the same as in Example 1 except that the Wurster  
insert was 1.9 cm (3/4 inch) from the bottom plate and the  
inlet temperature was in the 70-74°C range.  
One thousand grams of <0.6mm>0.25mm (-30 +60 mesh) non-pareils  
(sugar crystals-sugar solution-starch-dextrin-glaze)  
10 was charged to the Uni-Glatt and fluidized. An  
aqueous enzyme slurry with 19% (w/w) dry solid having  
activity of 643.8 DAPU/g and 252,632 MWU/g (modified  
Wohlgemuth unit per gram) was fed into the dryer for  
coating at the rate of 12 ml/min. A total of 2000 g  
15 of enzyme slurry containing 380 g of enzyme solid was  
used.

The enzyme coated particles were further coated  
with 146 g of a 50% (w/w) solution containing 73 g of  
polyethylene glycol (MW 4000) in water at a feed rate  
20 of 12 ml/min. and an inlet air temperature of  
50-54°C.

A final total of 1453 g of dust free particles  
was harvested with a final activity of 846 DAPU/g and  
339,045 MWU/g as determined by the Wohlgemuth Alpha-  
25 Amylase Procedure, Miles Laboratories, Inc. Enzyme  
Approved QA Procedure #ME400.03. This test resulted  
in a recovery of 100% of mass balance yield and a  
97.5% recovery of enzyme activity.

1

EXAMPLE 3

Pilot Scale Fluid Bed Coating  
of Alkaline Protease

Fifty kilograms of  $\langle 0.6\text{mm} \rangle 0.25\text{ mm}$  (-30 +60 mesh) NaCl salt  
5 crystals were charged and fluidized in a Glatt fluid  
bed dryer model GPCG-60 with an 45,72 cm (18") Wurster insert.  
Aqueous enzyme slurry at 813.5 DAPU/g with an 18% dry  
solid content was fed into the dryer for coating at  
the rate of 125 ml/min. for the first 10 minutes, 200  
10 ml/min. for 110 minutes, 300 ml/min. for 40 minutes,  
and 450 ml/min. for 20 minutes for a total of 6.858  
kg enzyme solid from 38.1 kg of slurry.

The enzyme coated salt crystals were further  
coated with 20.3 kg of a solution containing 6.1 kg  
15 (30% w/w) Iconol NP-100 in water at the feed rate of  
125 ml/min. for 80 minutes, 167 ml/min. for 30  
minutes, and 227 ml/min. for 22 minutes.

A final total of 61.1 kg dust free particles  
were harvested with a final activity of 354 DAPU/g.  
20 This experiment resulted in a 99.5% mass balance  
yield and 72.2% enzyme activity yield.

The GPCG-60 fluidized bed dryer is a production  
model fluid bed spray coater very similar in design  
to the Uni-Glatt except that it has a proportionally  
25 taller expansion chamber. It was operated under the  
following conditions:



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- |   |                                   |   |   |
|---|-----------------------------------|---|---|
| 1 | Air Regulation Flap               | : | Varies  |
|   | Partition Height                  | : | 2,54 cm (1 inch) Clearance from<br>bottom plate |
|   | Nozzle Size                       | : | 1.8 mm  |
|   | Inlet Air Temperature             | : | 70-74°C and 50-54°C                             |
| 5 | Outlet Air Temperature            | : | 29-32°C   |
|   | Angle Setting                     | : | 6 mm  |
|   | Atomization Air Pressure          | : | 4 Bar   |
|   | 45,72 cm (18 inch) Wurster Insert |   |   |

EXAMPLE 4

10      Pilot Scale Fluid Bed Spray Coating  
         of Alkaline Protease

Forty and seventeen one-hundredths kilogram of  
<0,6 mm >0,25 mm (-30 + 60 mesh) non-pareil (sugar crystals-sugar  
solution-starch-dextrin) was charged and fluidized in  
15 a GPCG-60 with exactly the same setup and operating  
conditions as in Example 3. Enzyme slurry at 813.5  
DAFU/g with 18% dry solid was fed into the dryer for  
coating at the rate of 75 ml/min. for the first 30  
minutes, increased from 75 to 400 ml/min. steadily in  
20 the following 110 minutes and then maintained at that  
rate for the remainder of the run. A total of 45.2  
kg of enzyme slurry was used to apply 8.136 kg of  
enzyme solid to the core particles.

The enzyme coated particles were further coated  
25 with 9.7 kg of a solution containing 2.91 kg (30%  
w/w) Iconol NP-100 in water at the flat feed rate of  
36 ml/min. for 81 minutes.

1 The particles were further coated with 11.6 kg  
of an aqueous solution containing 3.48 kg  $TiO_2$  (30%  
w/w) and 1.39 Iconol NP-100 (12% w/w) at the feed  
rate of 50 to 55 ml/min. for 214 minutes.

5 A final total of 55.2 kg was harvested with a  
final activity of 646 DAPU/g. This test resulted in  
97.6% mass yield and 97% activity yield.

EXAMPLE 5

Pilot Scale Fluid Bed Downward Spray  
Coating of Alkaline Protease

10

In this experiment a GPCG-5 fluidized bed dryer  
manufactured by Glatt Air Techniques with a single  
air atomized Schlick nozzle, as was the case in the  
previous examples, located concentrically 35.83 cm (14.5 inches)  
15 high from the bottom of the product bowl was used.  
Core material particles were fluidized by the inlet  
air to a height of 15.24 cm to 30.48 cm (6 to 12 inches) above the  
nozzle which enabled the coated particles to become dry  
before falling back down onto the product bowl. This

20 operation mode minimizes particle agglomeration.  
Ten kilograms of salt crystals were charged to  
the product bowl of the fluidized bed reactor and  
fluidized with 70°C inlet air to a product  
temperature of 45°C, as determined by a probe in the  
25 bed, whereupon enzyme slurry was sprayed into the  
area of fluidized core material. The slurry, which  
contained 16% dry solid with an enzyme activity of  
434.7 DAPU/g, was fed at a steady rate of 190 g/min.

- 1 The product temperature was consequently maintained at a steady range of 34-38° under these inflow and outflow conditions.

The enzyme coated particles were further coated with 2.6 kg of an aqueous solution containing 50% (w/w) Iconol NP-100 and 50% water. The solution was atomized at a spray rate of 70 g/min, at 50°C inlet temperature. Holding the inlet air at 50°C resulted in a product temperature of 37 to 41°C.

- 10 A final weight of 12.7 kilograms of dust free particles was harvested with a final activity of 305.9 DAPU/g. This test resulted in a 99.7% mass balance yield without activity loss.

#### GPCG-5 Operating Conditions:

15	Air Regulation Flaps	:	Inlet 100% Outlet 38%
	Atomization Air Pressure:	:	4 Bar
	Nozzle Size	:	1.2 mm
	Angle Setting	:	4.0 mm
	Inlet Air Temperature	:	70°C and 50°C
20	Product Temperature	:	34-41°C
	Air Inlet Filter Pressure	:	50 mm H <sub>2</sub> O
	Product Bed Pressure:	:	30 mm H <sub>2</sub> O
	Exhaust Air Filter Pressure	:	150 mm H <sub>2</sub> O

WHAT IS CLAIMED IS:

- 1        1. A method for the production of dust free  
enzyme containing particles which comprises the steps  
of:
  - 5            a) introducing a particulate, hydratable core  
material into a fluid bed dryer and main-  
taining the core particles suspended in the  
dryer's reaction chamber;
  - b) providing an aqueous slurry of a water  
soluble or dispersible enzyme and applying  
10           the enzyme to the surface of the core  
particles by spraying the slurry onto them  
while they are suspended in the reaction  
chamber to leave residual, dried enzyme  
coated on the core particles in an amount  
15           sufficient to provide the desired enzyme  
activity; and
  - c) spraying a solution or dispersion of a  
macro-molecular, film-forming, water  
soluble or water dispersible coating agent  
20           onto the enzyme coated core material while  
it is still suspended in the reaction  
chamber and drying the solvent to leave a  
continuous layer of the film-forming  
material on the enzyme coated core particle  
25           to provide the desired dust free enzyme  
containing particle.

2. The method of Claim 1 wherein the core material has a particle size of from 150 to 2,000 microns in its longest dimension.

3. The method of Claims 1 or 2 wherein the core particle is clay, a non-pareil, agglomerated potato starch, particulate salt, agglomerated trisodium citrate, pan crystallized NaCl flakes, bentonite granules or prills, bentonite/kaolin/diatomaceous earth disk pelletized granules or sodium citrate crystals and the enzyme is protease, an amylase or a lipase.

4. The method of any of the Claims 1 to 3 wherein the enzyme slurry contains 15% to 30% solids (w/w) of which 100% to 30% is enzyme and has a viscosity of 10 to 5,000 cps at room temperature.

5. The method of any of the Claims 1 to 4 wherein the film-forming material is a fatty acid ester, an alkoxyated alcohol, a polyvinyl alcohol, or an ethoxylated alkylphenol, preferably a polyethylene glycol having a molecular weight of from 1,000 to 8,000, a linear alcohol alkoxylate having a molecular weight of from 1,450 to 2,670, a polyvinyl pyrrolidone having a molecular weight of from 26,000 to 33,000, polymeric nonylphenyl ethoxylates having a molecular weight of from 1,975 to 4,315 or a polymeric dinonylphenyl ethoxylate having an average molecular weight of 6,900.

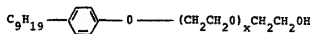
6. The method of any of the Claims 1 to 5 wherein there is applied sufficient enzyme and film-forming material to provide a total dry weight gain of 25% to 55% based on the weight of the core particles.

5 7. An enzyme containing particle which comprises:

- a) a particulate, highly hydratable core which is 150 to 2,000 microns in its longest dimension;
- 10 b) a uniform layer of enzyme around the core particle which amounts to 10% to 35% by weight of the weight of the core particle; and
- 15 c) a layer of a macro-molecular, film-forming, water soluble or dispersible coating agent uniformly surrounding the enzyme layer wherein the weight of the combination of enzyme and coating agent is from 25% to 55% of the weight of the core particle.

20 8. The particle of Claim 7 wherein the enzyme layer contains up to 70% by weight of one or more metallic salts, binders, plasticizers or fragrances.

25 9. The particle of Claims 7 or 8 wherein the enzyme is alkaline protease and the film-forming material is characterized by the formula



where X ranges from about 40 to 100.

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10. The particle of any of the Claims 7 to 9 wherein the core is a non-pareil having a sugar crystal core enclosed in layers of corn starch which is coated with a layer of dextrin.